Standard Operating Procedure for the Determination of Polycholrinated Biphenyls (PCBs) in Drinking Water by Perchlorination to Decachlorobiphenyl (DCBP) and Analysis by Gas Chromatography with an Electron Capture Detector

1.0 Scope and Application

1.1 This procedure may be used for screening finished drinking water, raw source water, or drinking water in any treatment stage for polychlorinated biphenyls (PCBs). This procedure is applicable to samples containing PCBs as single congeners or as complex mixtures such as weathered, intact, or mixtures of commercial Aroclors. The procedure is incapable of identifying the parent PCBs because the original PCBs are chemically converted to a common product, decachlorobiphenyl (DCBP).

1.2 This procedure is primarily designed to function as a pass/fail test for decachlorobiphenyl at 0.5 ug/L. However, it will accurately measure DCBP from the method detection limit (MDL) to 5.0 ug/L. It is prone to false positive interferences and can result in a calculated weight of PCBs significantly greater than that of PCB originally present in the sample. If DCBP is detected at 0.5 ug/L or above, then an approved method for the analysis of PCBs should be used to accurately identify the source and measure the concentration of the PCBs.

1.3 PCBs are converted to decachlorobiphenyl on a mole for mole basis. Converting decachlorobiphenyl concentrations back to the original PCB concentration is beyond the scope of this method.

2.0 Summary of Method

About 1 liter of a water sample is placed into a separatory funnel and extracted with methylene chloride. The extract is dried, concentrated, and the solvent is exchanged to chloroform and reduced to a small volume (approximately 100 uL). The extract is then subjected to a perchlorination reaction with antimony pentachloride ($SbCl_5$) in the presence of an iron catalyst and heat to convert any polychlorinated biphenyls to decachlorobiphenyl. The extract is acidified and extracted with hexane. After purification, an aliquot of the extract is injected into a gas chromatograph equipped with an electron capture detector for separation and measurement. The gas chromatograph is calibrated using decachlorobiphenyl as the standard.

3.0 Definitions

3.1 Calibration Standard (CAL): A solution of decachlorobiphenyl used to calibrate the electron capture detector.

3.2 Laboratory Reagent Blank (LRB): An aliquot of reagent water that is treated as a sample. It is exposed to all glassware, apparatus, method solvents and reagents. The extract is concentrated to the final volume used for samples and is analyzed the same as a sample extract.

3.3 Laboratory Fortified Sample Matrix (LFM): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.4 Fortified Reagent Water: Laboratory reagent water fortified with varying quantities of a standard PCB solution to determine if the method adequately recovers the polychlorinated biphenyls from a clean water matrix and quantitatively converts them to decachlorobiphenyl.

3.5 Quality Control Sample: A sample containing known concentrations of analytes that is analyzed by a laboratory to demonstrate that it can obtain acceptable identifications and measurements with procedures to be used to analzye environmental samples containing the same or similar analytes. The anlayte concentrations are known by the analyst. Preparation of the QC sample by an outside source is highly desirable.

4.0 Interferences

4.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing equipment. Laboratory reagent blanks (LRBs) are analyzed routinely to demonstrate that these materials are free of interferences under the analytical conditions used for samples.

4.2 To minimize interferences, glassware (including sample bottles) should be meticulously cleaned. As soon as possible after use, rinse glassware with the last solvent used. Then wash with detergent in hot water and rinse with tap water followed by deionized water. Drain dry and rinse with acetone followed by hexane. Before using, it would be advisable to rinse each piece with an appropriate solvent.

4.3 In addition to PCBs, several compounds and classes of compounds will form DCBPP with varying yields when extracted and perchlorinated according to this procedure. Based upon a literature search, such compounds include biphenyl, polyhalogenated biphenyls, hydrogenated biphenyls, and polyhalogenated terphenyls. If such compounds are present in the extract, false positive or positively biased data will be generated.

5.0 Safety

5.1 Methylene chloride is described in the procedure as the extraction solvent and chloroform is described in the procedure as the solvent for the perchlorination reaction. Chloroform and methylene chloride have been tentatively classified as known or suspected human or mammalian carcinogens. Hexane, hexane + 15% methylene chloride or hexane + 15% ethyl ether may be substituted for methylene chloride to minimize laboratory personnel exposure to methylene chloride. Other less toxic solvents were evaluated for replacing chloroform in the perchlorination reaction but were found to be unsuitable. The toxicity or carcinogenicity of the remaining chemicals used in this method has not been precisely defined. Therefore, each should be treated as a potential health hazard, and exposure should be reduced to the lowest feasible level. A reference file of material safety data sheets is on hand for chemicals used in the laboratory.

5.2 Polychlorinated biphenyls have been classified as known or suspected human or mammalian carcinogens. Primary standards of these compounds should be prepared in an area specifically designed to handle carcinogens. Primary standards are available from commercial vendors.

5.3 Antimony pentachloride ($SbCl_5$) is a corrosive reagent that reacts violently with water. This compound must be used with extreme caution. All operations involving the pure reagent must be performed in a hood because appreciable quantities of volatile, potentially harmful materials will be lost to the atmosphere.

5.4 The perchlorination reaction described in this procedure requires that the sample extract be heated to 205^0C for about 30 minutes while hermetically sealed in a glass test tube. The solvents and volumes of reagents described in the procedure should be carefully reproduced; otherwise dangerous pressures may be generated during perchlorination. The following safety precautions are strongly recommended.

5.4.1 Use only the prescribed perchlorination glassware and visually check for flaws such as chips, cracks, strains, or scratches. Discard if any

abnormalities are noted.

5.4.2 After cooling, the perchlorinated product is still under slight pressure and should be carefully vented in a hood.

5.4.3 The neutralization of the antimony pentachloride involves an exothermic reaction and should be performed in a hood.

5.4.4 An explosion shield should be used during the perchlorination and neutralization procedures along with additional eye protection such as an 8 inch face shield.

5.5 Storage, labeling and disposal of PCBs must conform to all applicable laws and regulations. See 40 CFR Part 761.60; .40; .45, 40 CFR Part 761, Polychlorinated Biphenyls (PCBs) Manufacturing, Processing, Distribution in Commerce and Use Prohibitions.

6.0 Equipment and Supplies

6.1 Sampling equipment

Water sample bottles: Meticulously cleaned 1 liter glass bottles fitted with Teflon-lined screw caps.

6.2 Glassware

6.2.1 Separatory funnel: 2 liter with Teflon stopcock.

6.2.2 200 mL Zymark flasks for the Turbovap concentrator.

6.2.3 Vials

6.2.3.1 Amber glass of suitable volume for storage of standards.

6.2.3.2 Amber autosampler screw cap or crimp cap.

6.2.4 Screw-cap culture test tubes: 100 mm x 13 mm i.d. Pyrex with a Teflon-lined screw cap.

6.2.5 Screw-cap graduated centrifuge tubes: 15 mL with Teflon-lined screw cap.

6.2.6 Disposable Pasteur pipettes.

6.2.7 Volumetric flasks: 5 mL, 10 mL, 100 mL with ground glass stoppers.

6.2.8 1 liter graduated cylinder.

6.3 Gas chromatograph system and columns

6.3.1 Programmable capillary column GC equipped with a splitless injector and a linearized electron capture detector capable of generating a linear response for decachlorobiphenyl from at least 0.005 to 1.0 ng injected. The column oven temperature programmer should have multi-ramp capabilities from at least 60^0C to 300^0C. An autoinjector is highly recommended.

6.3.2 A Lab Systems X-Chrom Chromatography Data System was available at the time this method was written. This chromatography data system can be used to automate the entire run of a set of standards and samples. Consult the data system reference manual to set up the data system to automate the run. Basically, a method file needs to be created to run the instrument and an analysis file needs to be created to acquire the data.

6.3.3 A Restec RTX-50 column which is a 30 meter, 0.25 mm ID column with a 0.25 um film thickness is currently used, however, any appropriate column will work. This column is crossbond 50% phenyl - 50% methyl polysiloxane. This column is used with a single taper liner in splitless mode. The GC parameters used with this column are as follows (set on the GC page of the chromatography data system method):

Column head pressure (set on GC)	10 psi
Maximum oven temperature	300^0 C
Equilibration time	0.25 Min.
Injector temperature	250^0 C
Detector temperature	300^0 C
Purge Valve (check the appropriate one)	
	On at 0.75 min, off at 0 min
Oven program	140^0 C, hold 1.00 min
	4^0C/min to 200^0C hold 0.0
	8^0C/min to 300^0C hold 5.67

The autosampler parameters used with this column are as follows (set on the autosampler page of the chromatography data system method):

Active Injector	Front

Sample Washes	3
Sample Pumps	3
Viscosity	0
Injection volume	1.00 uL
Solvent A Washes	3
Solvent B Washes	3

The acquisition parameters used with this column are as follows (set on the acquisition page of the chromatography data system method):

Minimum peak width	2.00 seconds
Acquisition rate	750 HZ
Acquisition time	1800 seconds
Post injection delay	0.00 seconds
Time between runs	0.00 seconds
Voltage offset	10.00 mv
Enable voltage offset	Check
Dual channel mode	Normal

The retention time for decachlorobiphenyl using the above conditions is about 25.9 minutes.

6.4 Miscellaneous equipment

6.4.1 Zymark Turbvap II concentrator.

6.4.2 Analytical balance.

6.4.3 Eppendorf pipet: adjustable volume.

6.4.4 Pierce Reacti Therm Heating Module with nitrogen blow-down head.

6.4.5 Tecator 1015 block digestion system (fill the cells with sand and insert the perchlorination tubes into the sand).

6.4.6 Thermometer to measure temperature of block digestor.

6.4.7 Safety shield to fit in front of the block digester in a fume hood.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Solvents: High purity hexane, chloroform, methylene chloride, toluene, and methanol.

7.1.2 Antimony pentachloride ($SbCl_5$): > 98% purity

7.1.3 Iron powder: 99.1% or greater purity.

7.1.4 Hydrochloric acid solution 1+1: Dilute one part concentrated hydrochloric acid with one part of deionized water.

7.1.5 0.1N sodium bicarbonate solution: Dissolve 0.84g of ACS reagent grade sodium bicarbonate ($NaHCO_3$) in deionized water, dilute to 100 mL and mix.

7.1.6 Reagent water: Reverse osmosis water passed through a Barnstead Nanopure water system.

7.2 Standards

7.2.1 Prepare a stock solution of Arochlor 1260 at 5.00 ug/uL in methyl alcohol or obtain a similar mixture from a certified source. This lab normally uses a 1000 ug/mL solution from Protocol.

7.2.2 Prepare a stock solution of decachlorobiphenyl at 1.00 ug/uL in hexane or obtain a similar mixture from a certified source. This lab normally uses a 1000 ug/mL solution from Protocol.

7.2.3 Prepare a PCB fortification solution by diluting an aliquot of the Arochlor 1260 stock solution in methyl alcohol to produce about 10 mL of a solution containing 10.0 ng/uL (100 uL to 10 mL). Store in a 50-90% filled glass vial with a Teflon-lined screw cap.

7.2.4 Calibration standards: This lab uses the following calibration standards prepared in hexane.

7.2.4.1 0.05 ug/mL: Transfer 10 uL of the 5 ug/mL stock standard to a GC sample vial and dilute volume to 1 mL with hexane.

7.2.4.2 0.10 ug/mL: Transfer 20 uL of the 5 ug/mL stock standard to a GC sample vial and dilute volume to 1 mL with hexane.

7.2.4.3 0.25 ug/mL: Transfer 50 uL of the 5 ug/mL stock standard to a

GC sample vial and dilute volume to 1 mL with hexane.

7.2.4.4 0.50 ug/mL: Transfer 100 uL of the 5 ug/mL stock standard to a GC sample vial and dilute volume to 1 mL with hexane.

7.2.4.5 1.00 ug/mL: Transfer 200 uL of the 5 ug/mL stock standard to a GC sample vial and dilute volume to 1 mL with hexane.

8.0 Sample Collection, Preservation, and Storage

8.1 Samples are collected in either 4 liter amber glass jugs with Teflon-lined screw caps or they are collected in 1 liter amber glass jars with Teflon-lined screw caps.

8.2 No chemical preservation reagents are recommended. Samples must be stored at 4^0C until analysis to retard microbial action.

8.3 Samples must be extracted within 14 days of collection. Extracts and perchlorinated extracts may be stored for up to 30 days if protected from solvent evaporation.

9.0 Quality Control

9.1 Initial demonstration of laboratory capability:

9.1.1 Reagent water fortified with Aroclor 1260 is recommended for this test.

9.1.2 Fortify four to seven 1 liter portions of reagent water with 50 uL of the 10 ug/mL PCB 1260 solution (Sect. 7.2.1). Extract and analyze the fortified water samples according to the procedure (Sect. 11).

9.1.3 Calculate the recovery according to the formula:

$$\% \text{ Recovery} = \frac{(\text{Total ng found in extract}) \times 100}{691}$$

$$\text{Where } 691 = (500 \text{ ng}) \times \frac{\text{mw DCBP (499)}}{\text{mw Aroclor 1260 (361)}}$$

9.1.4 Determine the average concentration and the relative SD of the measurements. Average recovery should be 100% $\pm$ 20% with a RSD of

< 10%.

9.2 Prepare and analyze a laboratory reagent blank with each set of 10 samples. Using 1 liter of reagent water, perform all steps in the analytical procedure using the same glassware, reagents, solvents, equipment and instrumentation that would be used for a sample. An acceptable reagent blank contains ≤ 0.025 ug/mL of decachlorobiphenyl. If the reagent blank results are unacceptable, systematically check solvents, reagents (particularly the antimony pentachloride and methylene chloride), apparatus and glassware to locate and eliminate the source of contamination before analyzing samples. Purify or discard contaminated reagents and solvents. If the reagent blank results are unacceptable, any associated samples will need to be reanalyzed.

9.3 Analyze at least one sample of fortified reagent water for each batch of 20 samples. Fortify the reagent water with the 10 ug/mL PCB 1260 solution, varying the quantity from batch to batch. Calculate recovery according to the formula in section 9.1.3. Maintain quality control charts of these data. Until sufficient data points are available (a minimum of 20-30), the recovery of the fortified sample should be equivalent to that in section 9.1.4.

9.4 Check for sample matrix effects by analyzing one laboratory fortified sample matrix for every 20 samples.

9.4 At least quarterly, analyze a quality control sample from an external source. If the measured analyte concentration is not of acceptable accuracy, check the entire analytical procedure to locate and correct the source of the problem.

10.0 GC Calibration and GC Analysis of Quality Control Checks and Samples

10.1 Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required intermittently throughout sample analysis as dictated by results of continuing calibration checks. After initial calibration is successfully performed, a continuing calibration check is required at the beginning and end of each set of samples or 8 hour period during which analyses are performed.

10.2 Set up the chromatography data system method and analysis files so that 1 uL of each calibration standard is injected into the GC. Follow the injection of standards with injections of a laboratory reagent blank, a quality control sample, a laboratory fortified blank, samples, and a fortified sample matrix. For an acceptable continuing calibration check, the 0.1 ng/uL calibration standard must

be analyzed after a series of samples or at least once after each 8 hours of operation. The response factor must be within ± 20% of the initial response factor or a new calibration curve must be generated. This laboratory normally analyzes a full set of calibration standards following the samples. Process the standards with the chromatography data system and evaluate the calibration curve for acceptability. The calibration curve and continuing calibration checks must be acceptable before processing the quality control checks and samples.

11.0 Procedure

11.1 Sample extraction and concentration of extract

11.1.1 Mark the sample meniscus on the side of the one liter sample bottle for later determination of sample volume and pour the entire sample into a 2 liter separatory funnel or measure 1 liter of sample from the 4 liter sample jug and transfer to the separatory funnel. Transfer a duplicate of the sample which is to be fortified and add the fortification solution to this sample.

11.1.2 For the laboratory reagent blank, fortified reagent water, and the quality control sample measure 1 liter of regent water and transfer to each of three separatory funnels. Add the appropriate volume of the PCB 1260 solution to one of the separatory funnels for the fortified reagent water, and add an appropriate volume of the quality control sample concentrate to one of the other separatory funnels.

11.1.3 Add 60 mL of methylene chloride to the 1 liter sample bottles (if used), seal, and shake 30 seconds to rinse the inner surface and transfer the solvent to the separatory funnel. Otherwise, add 60 mL of methylene chloride directly to each separatory funnel and extract the samples by shaking the funnel for 2 minutes with periodic venting to release excess pressure. Separatory funnels should be used in a fume hood and venting must be done in a fume hood. Wait at least 10 minutes for the organic layer to separate from the water phase. If an emulsion forms between layers and is more than one-third the volume of the solvent layer, use mechanical techniques (such as stirring, filtration of emulsion through glass wool, or centrifugation) to complete the phase separation. Dry the interior of the drain tube of each separatory funnel with a kim wipe. Drain the methylene chloride extract into a Zymark tube. Add a second 60 mL portion of methylene chloride to the sample bottle or separatory funnel and repeat the extraction procedure a second time, combining the extracts

in the Zymark tube. Perform a third extraction in the same manner. The Zymark tubes can be placed in the Zymark concentrator to begin evaporation of the methylene chloride while the subsequent extractions are being performed.

11.1.4 Continue concentrating the extract down to about 1 mL in the Zymark tubes. The Zymark concentrator will beep when the volume reaches about 1 mL.

11.1.5 Quantitatively transfer the extract to a 100 mm x 13 mm i.d. screw cap test tube using a disposable transfer pipette. Rinse the Zymark tube two or three times with approximately 1 mL of chloroform and transfer the rinse to the test tube.

11.1.6 Concentrate the extract in the test tube to about 0.1 mL (about the volume of one drop of water) by directing a stream of nitrogen into the test tube while warming the base of the test tube in a Pierce Reacti Therm heating module or in a 50^0 C water bath. **Do not allow to go to dryness!**

11.1.7 Add an additional 2 mL of chloroform and again concentrate to 0.1 mL using the nitrogen blow-down technque.

11.2 Perchlorination

11.2.1 Preheat the block digester to 205 ± 5^0 C. Dial set at approximately 4.75.

11.2.1 Add 100 mg of iron powder (in drawer on east wall) to the extract in the tube.

11.2.2 Then, using a disposable pipette, carefully add 25 drops of antimony pentachloride (underneath solvent rinse hood) to the extract in the tube.

11.2.3 Burrow each tube into the sand in a separate cell in the 205^0 C block digester and heat for a minimum of 30 minutes but do not exceed 45 minutes. **Perform the reaction in the hood behind an explosion shield!**

11.2.4 Allow the mixture to cool to room temperature.

GOOD PLACE TO STOP (If needed).

11.3 Neutralization, extraction of decachlorobiphenyl, and clean up

11.3.1 Carefully open the tubes in a hood. (The contents will be under a slight pressure).

11.3.2 Slowly add 0.5 mL of 1+1 hydrochloric acid to each tube. An Eppendorf pipette set to deliver 500 uL works well for this. Do this in a fume hood. **Caution: the remaining antimony pentachloride will react exothermally with the HCl!** If a white precipitate is present, add additional hydrochloric acid solution until it dissolves.

11.3.3 Add 2.0 mL of hexane to the contents of the test tube, seal and shake for 2 minutes. Allow the two phases to separate and decant the top layer into a 5.0 mL volumetric flask. Reextract the mixture two additional times, first with 2.0 mL of hexane, then with 1.0 mL of hexane, adding the extracts to the 5.0 mL volumetric flask. Carefully adjust the volume to 5.0 mL using hexane.

11.3.4 Add 4 mL of 0.1N $NaHCO_3$ to a 15 mL graduated centrifuge tube with a Teflon-lined screw cap. Pour the contents of the 5 mL volumetric flask into the test tube but do not rinse the flask with additional solvent. Seal and shake for 1 minute and allow the two phases to separate.

11.3.5 Transfer the top layer into a second 15 mL graduated centrifuge tube using a disposable transfer pipette. Add 4 mL of reagent water, seal and shake for 1 minute.

11.3.6 Transfer the hexane (top) layer into autsoampler vials and seal for GC analysis.

11.3.7 Analyze on the gas chromatograph according to section 10.2.

11.3.8 Process the quality control checks and samples with the chromatography data system and evaluate the results of the quality control checks. All quality control check results must be acceptable before results for any associated samples can be reported. All sample concentrations must be bracketed by the calibration curve and must be within the linear dynamic range of the detector. Samples that fall outside the linear dynamic range due to excessive concentration must be reanalyzed after appropriate dilution if accurate values for DCBP are required.

12.0 Data Analysis, Calculations, and Reporting Results

12.1 Calculations

12.1.1 Determine the original sample volume, if the sample was collected in a 1 liter bottle, by refilling the sample bottle with water to the mark and transferring the liquid to a 1000 mL graduated cylinder. Record the sample volume to the nearest 5 mL.

12.1.2 The chromatography data system can calculate the concentration of the decachorobiphenyl found in each extract in the same units as the standards (ng/uL). The chromatography data system user equations can be used to calculate results in the sample in ng/L or ug/L or the following equation can be used external to the system:

$$\text{Sample concentration ng/L} = \frac{(\text{Concentration ng/uL})\ (5000)}{\text{Volume of sample (L)}}$$

Where:

5000 = final volume of extract in uL

Volume of sample (L) = volume of sample extracted in liters

12.1.3 Calculations should use all available digits of precision. Do not subtract method blanks from the sample data unless otherwise required in the procedure.

12.2 Reporting results

12.2.1 Results should be reported in ug/L unless otherwise specified.

12.2.2 Final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty). Experience indicates that three significant figures may be used for concentrations above 99 ug/L, two significant figures for concentrations between 0.1-99 ug/L and one significant figure for lower concentrations.

13.0 Method Performance

To obtain single-laboratory accuracy and precision data for method analytes when the EPA method was developed, seven 1 liter aliquots of chlorinated tap water, ground water and river water were fortified with 500 ng of PCBs from several sources. The samples were extracted, perchlorinated and analyzed according to section 11. Tables 1 and 2 list the resulting data.

14.0 Pollution Prevention

14.1 Methylene chloride and chloroform are evaporated from the extracts and during the solvent exchange prior to perchlorination so there is no remaining waste methylene chloride or chloroform.

14.2 Only small amounts of antimony pentachloride are used so the quantity of waste generated is minimal.

15.0 Waste Management

15.1 Disposal of PCBs must conform to all applicable laws and regulations. See 40 CFR Part 761.60; .65; .40; .45 of 40 CFR Part 761, Polychlorinated Biphenyls (PCBs) Manufacturing, Processing, Distribution in Commerce and Use Prohibitions.

15.2 Waste remaining in the test tubes after extraction of the perchlorinated samples with hexane should be stored in an appropriate, labeled waste container for proper disposal.

15.3 For further information on waste management consult *The Waste Management Manual for Laboratory Personnel* and *Less is Better: Laboratory Chemical Management for Waste Reduction*, both available from the American Chemical Society's Department of Government Relations and Scienc Policy, 1155 16th Street N.W., Washington, D.C. 20036.

16.0 References

16.1 EPA Method 508A, *Screening for Polychlorinated Biphenyls by Perchlorination and Gas Chromatography*, Revision 1.0 (1989), *Methods for the Determination of Organic Compounds in Drinking Water,* EPA/600/4-88-039, December 1988, Revised, July 1991.

16.2 LabSystems Xchrom Reference Manual.

16.3 *HP 5890 A Gas Chromatograph Reference Manual Volumes I and II*, Hewlett Packard Company, Avondale, Pennsylvania.

16.4 *HP 7673 Automatic Sampler Operating and Service Manual,* Hewlett Packard

Company, Avondale, Pennsylvania.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

TABLE 1
Splitless Capillary Column Single Laboratory Accuracy and Precision
for Fortified Tap Water

Source of PCBs	MDL ug/L	Concentration (ug/L)	Accuracy[ae] %	Precision[ae] RSD, (%)
2-Chlorobiphenyl	0.08	0.50	85; (96)[b]	5.0; (9.9)[b]
Arochlor 1221	0.14	0.50	99	8.4
Arochlor 1232	0.23	0.50	124	11.3
Arochlor 1242	0.21	0.50	82	13.1
Arochlor 1248	0.15	0.50	136	8.6
Arochlor 1254	0.14	0.50	122; (137)[c]	6.4; (7.6)[c]
Arochlor 1260	0.14	0.50	113; (96)[b] 6.5;	(6.9)[b]
Biphenyl[d]		0.50	109; (75)[c] 4.8;	(5.8)[c]

[a]Data corrected for source water background. Average value over study = 0.11 ug/L.

[b]Data collected by on-column capillary column GC.

[C]data collected by packed column GC.

[d]Potential method interference compound.

[e]Fortified matrix effect bias

TABLE 2
Splitless Capillary Column Single Laboratory Accuracy and Precision
for Raw Source Water

Raw Source Water	Source of PCBs Arochlor	Concen-tration (ug/L)	Extraction Solvent	Source Water Background (ug/L)	Accuracy (%)	Precision RSD (%)
Ohio River	1221	0.50	CH_2Cl_2	0.54	114	8.4
Spring	1260	0.50	CH_2Cl_2	0.19	101	7.9

Ohio River	1221	0.50	Hexane	0.16	123	7.5
Little Miami River	1260	5.0	Hexane	0.14	91	5.8
Ohio River	1260	5.0	Hexane	0.29	100	5.4